



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No. : 09/933,535 Confirmation No. 5814
Applicant (s) : Edward O. Shaffer II et al.
Filed : August 20, 2001
TC/A.U. : 2811
Examiner : Lisa A. Kilday
Title : ORGANOSILICATE RESINS AS HARDMASKS FOR ORGANIC
POLYMER DIELECTRICS IN FABRICATION OF
MICROELECTRONIC DEVICES

Docket No. : 60393B
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DATE OF SIGNATURE

*Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450*

Sir:

DECLARATION OF Lynne Mills
SUBMITTED UNDER 37 CFR §1.132

1. I am a named coinventor of the above identified Application.
2. I am a Research Specialist in the Advanced Electronic Materials business at The Dow Chemical Company. I have worked for The Dow Chemical Company since 1989.
3. I received my M.S. in ceramic science from Rutgers University.
4. The tests performed in this declaration were performed by Shellene Thurston, a contract employee at Dow.

5. Phenyltrimethoxysilane (PT) and vinyltrimethoxysilane (VT) and were added to a glass reaction vessel in mole ratios of PT/VT or 15/85, 30/70, 50/50, 75/25, and 95/5. Acetic acid was added to the vessel over thirty minutes. The vessel was heated and the reaction proceeded for the times set forth in the table to provide the molecular weights set forth in the table.

6.

PT/VT mole ratio	15/85	30/70	50/50	75/25	95/5
B-Stage hrs	20	20	31	45	71.5
Molecular Weights	4900	5000	5142	3686	4085
Cure temperature used to reach film retention > 98% w/o BTAC	<250C	275C	325C	325C	350C
Adhesion as measured by m-ELT (mPa-m ^{1/2})	0.39	0.36	0.36	0.3	0.22
Adhesion as measured by dry tape peel	5B	5B	5B	5B	4B
Adhesion as measured by PCT Tape Peel	5B	5B	5B	5B	4B

7. The oligomers were diluted with PGMEA to a desired percent solids and spin coated onto a substrate. After spin coating, the films were hot plate baked at the temperatures set forth in the table until at least 98% film retention was obtained. Film retention was determined by measuring the thickness of the film after hot plate bake, rinsing the film with approximately 3 ml of PGMEA while spin coating, drying the film at 80 C on a hot plate and than remeasuring the film thickness. Film retention was calculated using the following formula:

$$(\text{Post-PGMEA Thickness}/\text{Initial Thickness}) * 100\%$$

As shown in the table, as the phenyl content increased, the temperature required to cure the films increased.

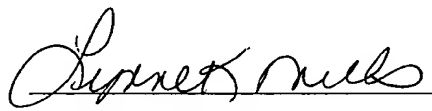
8. A second set of samples were than prepared by diluting the concentrate oligomer samples to 0.75% solids. These samples were spin coated as described above and cured using the temperatures used to reach 98% film retention. After curing, nominal 600 nm SiLK* semiconductor dielectric (a trademark of The Dow Chemical Company) films were spun on top of the silsesquioxane films as described above

and hot plate baked at 305 C for 2 minutes. These stacked films were than cured at 400 C for 30 minutes in a nitrogen purged furnace.

9. After cure, the adhesion of the stacked films were measured using the modified edge lift off test as taught in U.S. Patent 6,790,792. The adhesion for samples with the low vinyl content of 5 mole percent was significantly lower than for the other samples. In addition, a dry tape peel test was performed on all of the stacked films. The stacks containing the 5% vinyl content films showed reduced tape peel adhesion compared to the other films.

The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the above-entitled patent application or any patent issuing thereon.

Date 11-11-04


Lynne K. Mills

* Trademark of The Dow Chemical Company